‘Testing’
Accelerating evaluations of new fusion materials

Bruce Pint

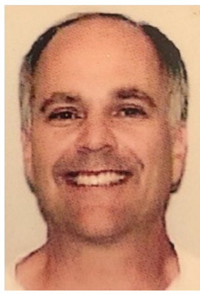
Materials Science & Technology Division
Oak Ridge National Laboratory
Oak Ridge, TN 37831-

Acknowledgments

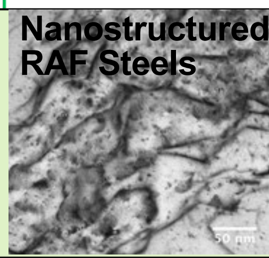
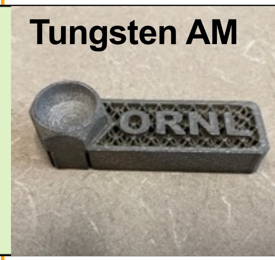
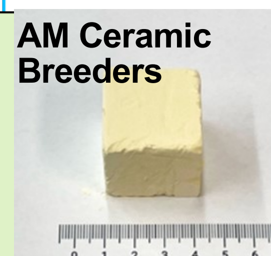
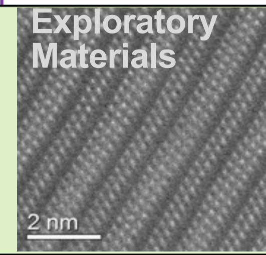
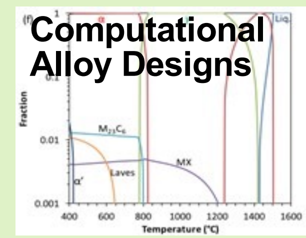
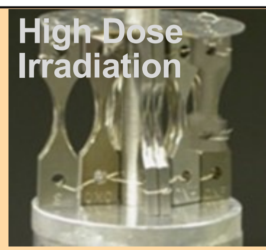
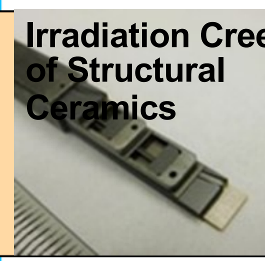
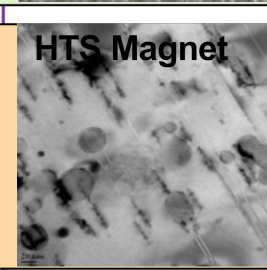
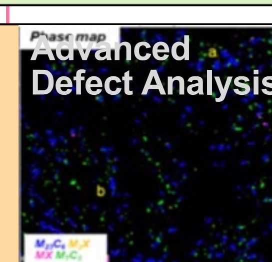
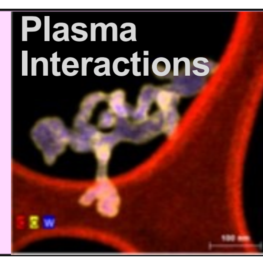
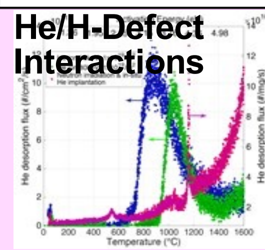
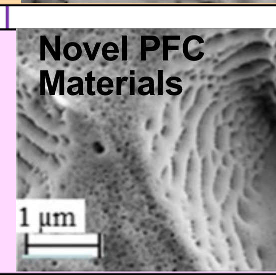
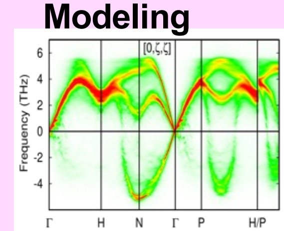
- Funding
  - U.S. Dept. of Energy, Office of Fusion Energy Sciences
- ORNL team
  - Yutai Kato, interim division director & fusion materials program manager
  - Chuck Kessel (retired ORNL, January 2023)
  - Paul Humrickhouse
  - Previous leaders: Everett Bloom, Steve Zinkle, Roger Stoller, Lance Snead
  - Dozens of collaborators
- Mentors: Jack DeVan, Jim DiStefano, Peter Tortorelli, Steve Pawel

DeVan 1929-2000

DiStefano 1935-2013



ORNL is a leader in fusion materials R&D in the DOE Office of Science

R&D items shown are examples from recent studies	Steels & Ferrous Alloys	Plasma Facing Component Materials	Structural Composites & Functional Ceramics	Novel & Emerging Materials	Advanced Characterization, Modeling & Computation
Materials Development	Nanostructured RAF Steels 	Tungsten AM 	AM Ceramic Breeders 	Exploratory Materials 	Computational Alloy Designs 
Effects of Nuclear Environment	High Dose Irradiation 	Tungsten Phase Stability 	Irradiation Creep of Structural Ceramics 	HTS Magnet 	Advanced Defect Analysis 
Specific & Integrated Fusion Environment	LM & Salt Compatibility 	Plasma Interactions 	He/H-Defect Interactions 	Novel PFC Materials 	Modeling 

Ye shall know the truth, and the truth shall make you mad. - Aldous Huxley

- TMS 2004 talk: Oxidation Resistance: One Barrier to Moving Beyond Ni-Base Alloys (B.A. Pint and I. G. Wright)
  - "Noburnium" won best poster award: the best materials stay on paper
- Mater. Sci. Eng. A 2006 paper: Oxidation Resistance: One Barrier to Moving Beyond Ni-Base Alloys (B. A. Pint, J. R. DiStefano & I. G. Wright)
- TMS 2009 talk: High-Temperature, Environmental-Resistant Coatings for Current and Future Alloys
- TMS 2014 talk: Beyond Ni-base Superalloys: The Environmental Resistance Barrier
- Mater. Sci. Tech 2014 paper: Critical Assessment 4: Challenges in developing high temperature materials (dedicated to J. R. DiStefano)
- **TMS 2021 talk**: Beyond Superalloys: An Efficient Strategy for Assessing Environmental Resistance (selected for TMS 75th anniversary)

2014 Thoughts: still seem spot on to me

- John Elliott's words from 1988 are in my head:
  - "that would only impress the uninformed"
- Research dollars are scarce + life is short
  - identify what is import for your work (and do it)
    - If ductility is an issue, measure it
    - If the coating protects from embrittlement, prove it
    - If neutron resistance is critical, then irradiate materials with neutrons
- Growing gap between research & application
  - development should be tied to reality
  - become educated about the key issues
  - ask someone in industry what will impress them
- Coatings should not be considered prime reliant
  - meaning: materials must be compatible with their operating environment
    - If a coating is needed for compatibility, that will always be a major design challenge

2014 talk focused on AFRL "TEST" concept

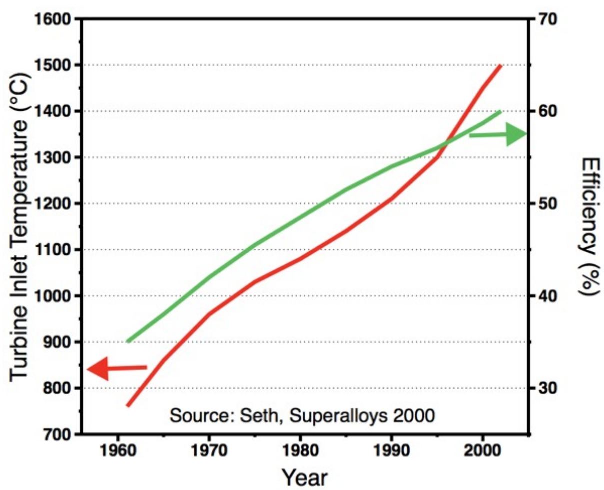
AFRL (Air Force Research Laboratory)

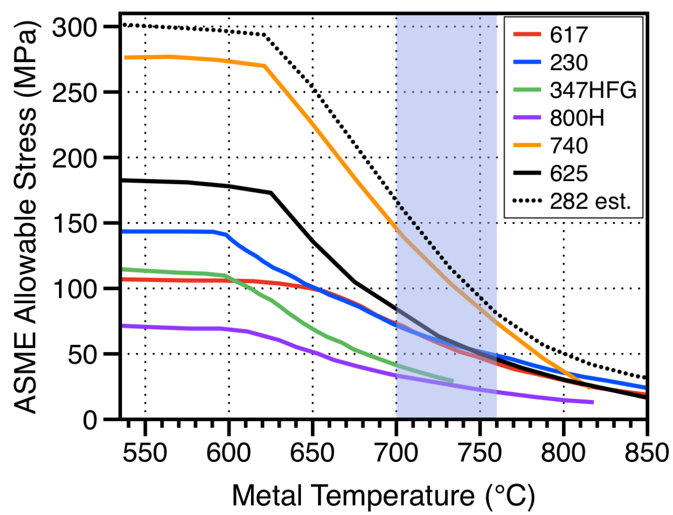
- Temperature
- Environment
- Stress
- Time
  - Let's say 25,000 h for fusion reactor (3 years)

The 2014 TEST slide ended with "testing is not the paradigm today"

What service temperatures are required?

- Most alloys lose their strength at 600°-800°C
- Most conventional alloys melt at <1500°C
- Most ceramics melt at <2000°C





“OK boomer”

Turk USC coal plant (AR)
2013: 607°C max. steam

330°C is hot for a nuclear reactor

Today's Theme B

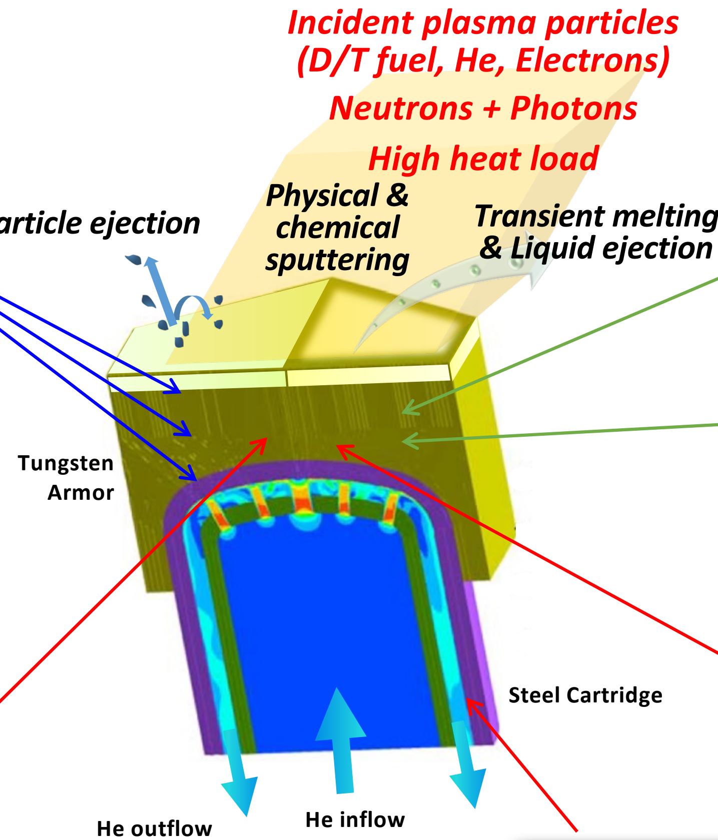
- goal of developing an integrated teaming environment to conduct high throughput materials discovery and experiments that systematically generate design data to enable the rapid deployment of new materials in commercial fusion power plants (FPP)
  - 1) assessing the feasibility and limits of machine learning (ML) or artificial intelligence (AI) tools to rapidly search for novel materials for fusion power plant applications
  - 2) identifying scalable and reliable manufacturing techniques that can integrate and support high throughput production of fusion material test samples and commercial components
  - **3) identifying key methods and challenges for high throughput material experiments to generate reproducible and repeatable data to rapidly verify material performance in fusion power plant conditions, and**
  - 4) identifying key features and interfaces for a fusion materials design data repository that can function as the central database for all stakeholders within the fusion community

Let's focus on materials for the blanket

- Blanket has three roles
  - Contain the plasma
  - Extract useful heat (i.e. make power)
  - Breed tritium (not going far without fuel)
- Materials needs are design dependent
  - Designs are variable so it is difficult to establish **universal metrics**
    - Perhaps a subject for discussion
  - Working list of properties for today
    - Neutron energy and flux
    - X-ray energy and flux
    - Time at temperature
    - Stress over time
    - Corrosion
    - Erosion

- Expensive!
- optics?
- Throughput…
- Throughput…
- No relevant fast test
- Concept-dependent

Combined Extremes: hard to think about one test/property #1 – Fusion Plasma Facing Components = Multi-Physics Interplay



Mechanical Integrity (Bulk, Surface & Interface)

Stress in Material

$$\sigma, \tau = f(T, \Delta T, \dot{T}, \cdots)$$

Strength of Material

$$\sigma_F, \tau_F = f(T, n, C_{He}, C_{Tr}, \cdots)$$

High Temperature and Gradient Effects

Temperature/Gradient

$$T, \Delta T = f(q''', K_{th}, \cdots)$$

Tritium Retention

Tritium Solubility

$$K_{Tr} = f(T, n, C_{He}, \cdots)$$

Tritium Permeation

Tritium Flux

$$\phi_{Tr} = f(T, \Delta T, n, C_{He}, C_{Tr}, \cdots)$$

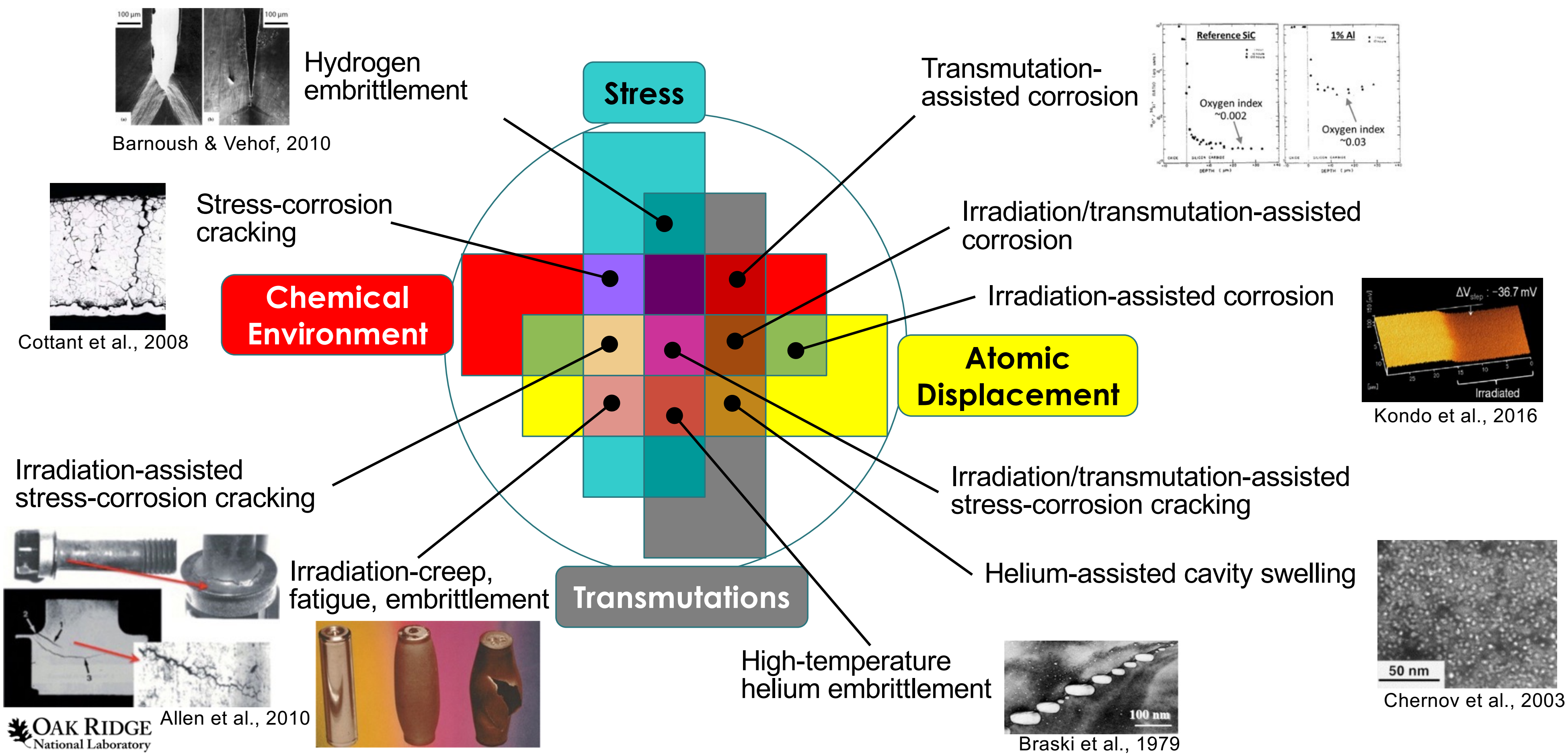
Heat Transfer (Bulk & Interface)

Thermal Conductivity

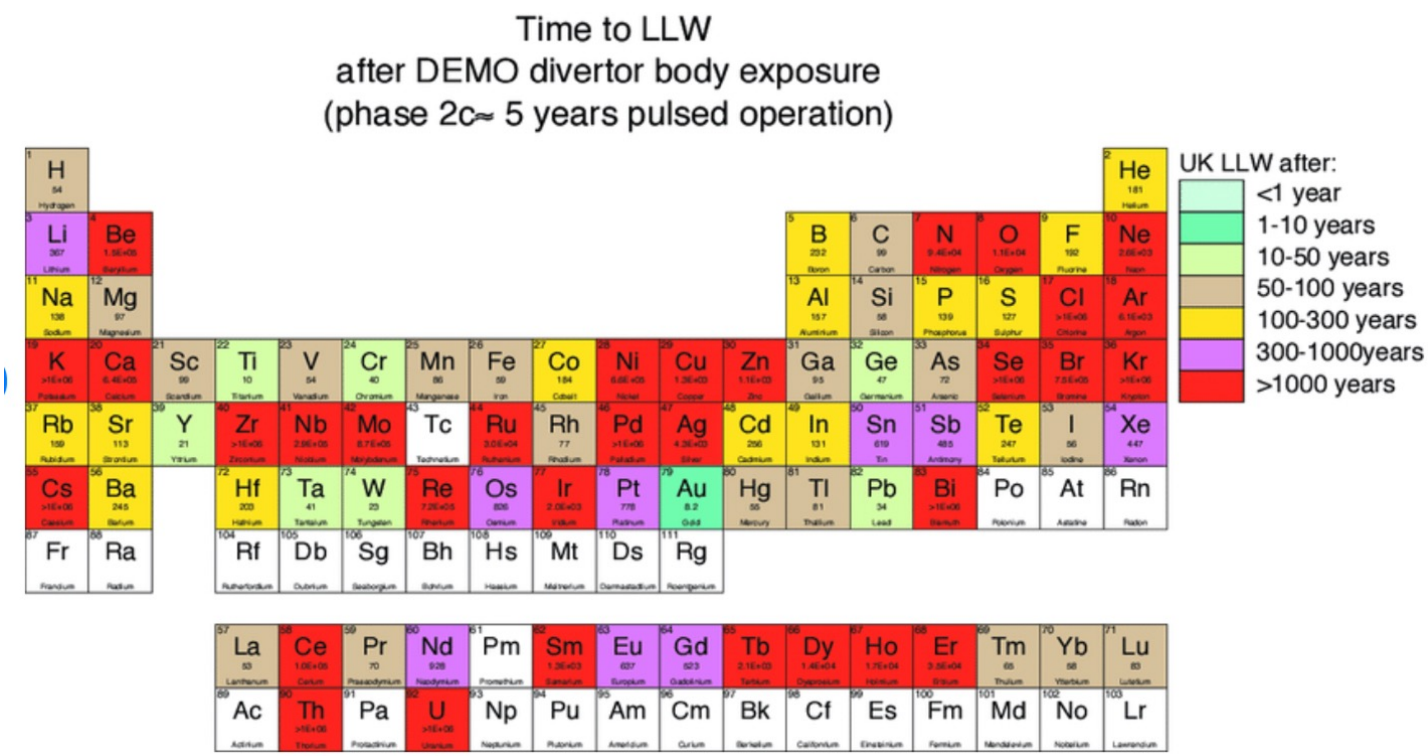
$$K_{th} = f(T, n, C_{He}, C_{Tr} \cdots)$$

Heat Removal by He Flow

Combined Extremes: hard to think about one test/property #2 – Stress Corrosion Cracking = Synergistic Effects



Good & bad elements for the fusion environment



Periodic table of the calculated time period that each element would require to decay to UK low level waste limits (<4 MBq kg −1 alpha radiation and <12 MBq kg −1 combined gamma and beta radiation) following exposure inside a DEMO fusion reactor. The component considered is the divertor over a time period equivalent to 5 years of pulsed operation. Reproduced from Gilbert et al. [15].

Fusion materials today

Three current materials

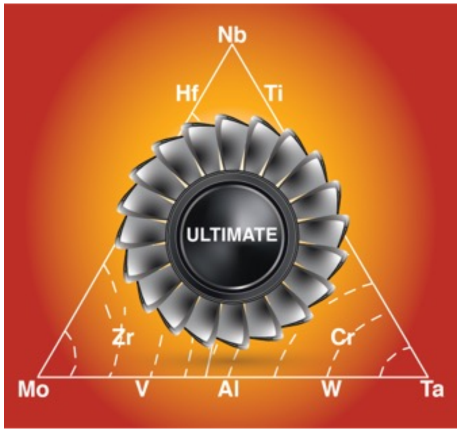
- RAFM (reduced activation ferritic martensitic) steel
  - All CSEF (Creep strength enhanced ferritic) steels similar to Grades 91, 92
    - CSEF: multi-billion $ industry
  - Fe-8Cr-2W (typical composition)
- SiC: SiC matrix + SiC fiber composites
  - Composite to improve toughness
  - Fabrication & design challenge
- V alloy: refractory metal
  - V-4Cr-4Ti was leading US composition
  - High O solubility: easily damaged

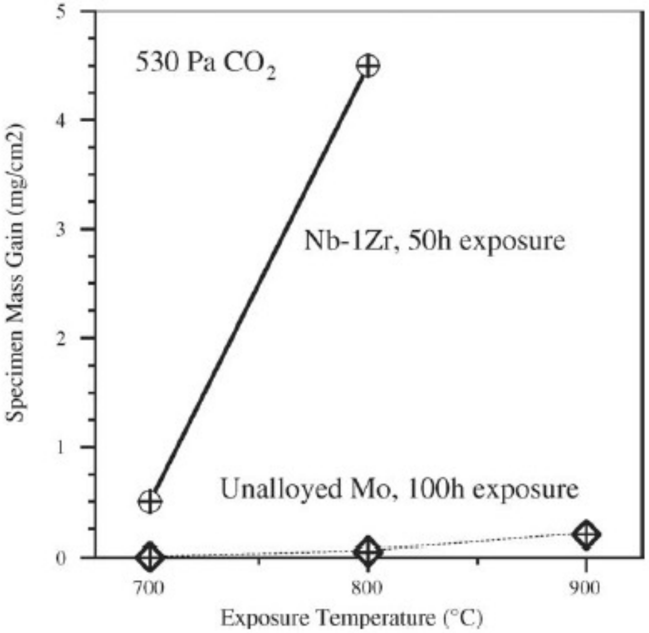
Common blanket concepts

- $^{6}Li + {}^{1}n \rightarrow {}^{4}He + T$
- Solid breeder
  - Li containing ceramics
  - design: when does T come out?
- Liquid breeder
  - Li (T_m=181°C)
  - Pb-Li (T_m = 235°C)
    - Eutectic with low Li activity
  - FLiBe (T_m = 459°C)
    - $2LiF\text{-}BeF_2$ molten salt
  - Either toxic or flammable…

Refractory metals are unusual

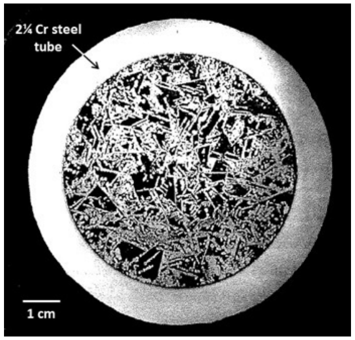
- Love the high melting points, but…
- Nb, Ta, V, Zr, Cr, Ti
  - High solubility for O
  - Easily embrittled!
  - Stable oxides but fast growth (except Cr)
- Mo and W
  - Low solubility for O
  - Unstable oxides (evaporate)
- Which problem do you want?
  - 1950’s: GE Mo-base, P&W: Nb-base
  - 1990’s: GE Nb-base, P&W: Mo-base





Liquid metal degradation modes: what are we afraid of?

- Wetting
  - Required for reactions to occur
    - SiC did not wet Pb-Li at 800°-1000°C
- Dissolution (and precipitation on cooling)
  - **Temperature limit not set by dissolution but by threat of plugging flow**
  - Ni high solubility in Pb (Fe and Cr also significant) and Li
  - Most oxides & SiC stable in Pb-17Li (Li activity is 10^{-3} in eutectic at 800°C)
  - Dissimilar material interactions
    - Fe in Pb-Li could react with C in SiC

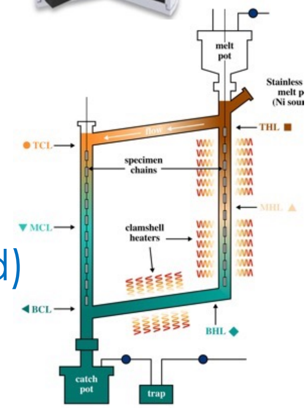


- Segregation
  - Embrittle alloy grain boundaries
  - Alter electrical properties (Li diffuses rapidly even at room temperature)
- Alloying between liquid and solid (showstopper)
- Similar dissolution issues for molten salts (FLiBe)

How do we assess liquid metal/molten salt compatibility? This strategy has been used for 70+ years

~$5K test

- Thermodynamics
  - First/rapid screening tool but data is not always available
- Capsule/crucible (screening test, one material per test)
  - Isothermal test, first experimental step
  - Prefer inert material and welded capsule to prevent impurity ingress
  - **Dissolution rate changes with time** & **No assessment of mass transfer**
- Thermal convection loop (TCL)
  - Flowing liquid metal by heating 1 side of “harp” with specimen chain in legs
  - Relatively slow flow and ~100°C temperature variation (design dependent)
  - Captures solubility change in liquid: dissolution (hot) and precipitation (cold)
    - Dissimilar material interactions between specimens and loop material



~$150K test

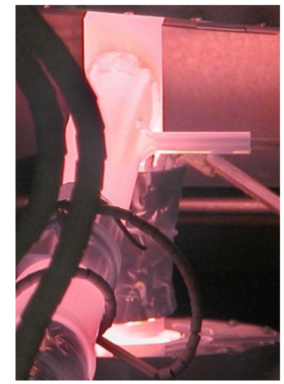
- Pumped loop
  - Most realistic conditions for flow
  - Historically, similar qualitative results as TCL at 10+X cost
  - Necessary progression for other aspects of LM blanket development
  - **Need results ASAP, including with magnets and radiation**

~$5M test

~$??M test

ORNL compatibility topics for fusion energy

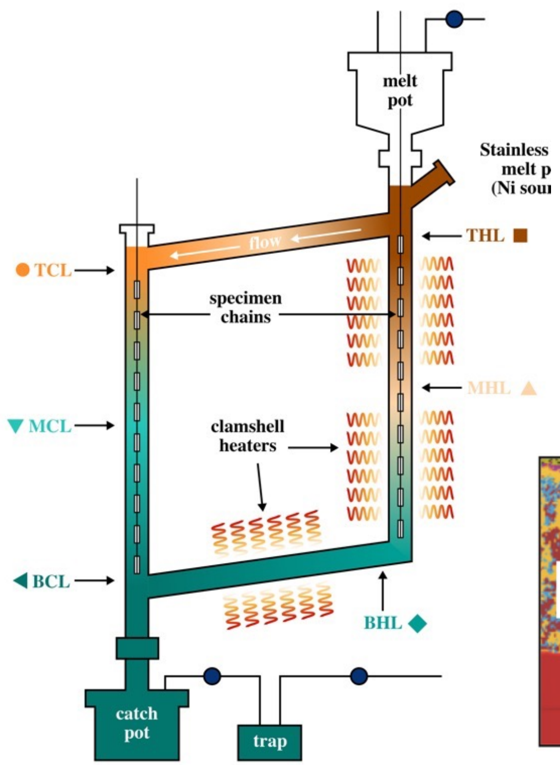
- 1992-1998: V-4Cr-4Ti oxidation in low pO_2 environment (vacuum/He)
  - Reaction kinetics on how fast V alloys embrittle
- 1998-2007: MHD coatings for V/Li blanket
  - Most insulating oxide candidates dissolve in Li
  - Spent ~5 years proving CaO dissolves (others advocated CaO)
  - Finished with 700°C V-4Cr-4Ti loop with PVD V/Y_2O_3 coatings



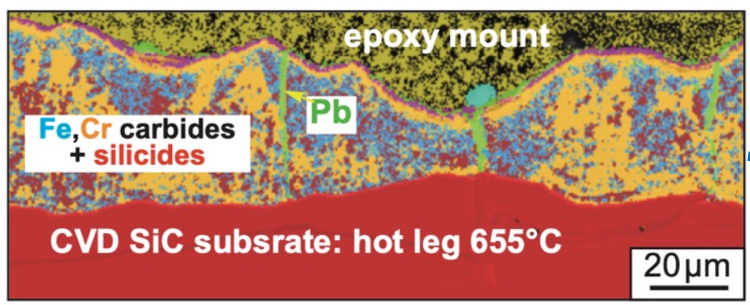
V-4Cr-4Ti loop at 700°C through chamber viewport

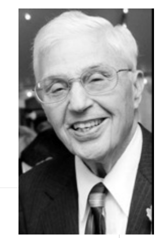
- 2003-present: Compatibility in Pb-Li
  - Started with SiC and composites (800°-1200°C capsules)
  - Wrought + ODS FeCrAl & FeCr alloys w/o Al-rich coatings (capsules)
  - Flowing experiments: monometallic (2014-2020) + multi-material (2021+)
    - Kanthal FeCrAlMo alloy APMT (A is for available)

Flowing loop tests with monolithic SiC & FeCrAl in Pb-Li Dissimilar material interaction

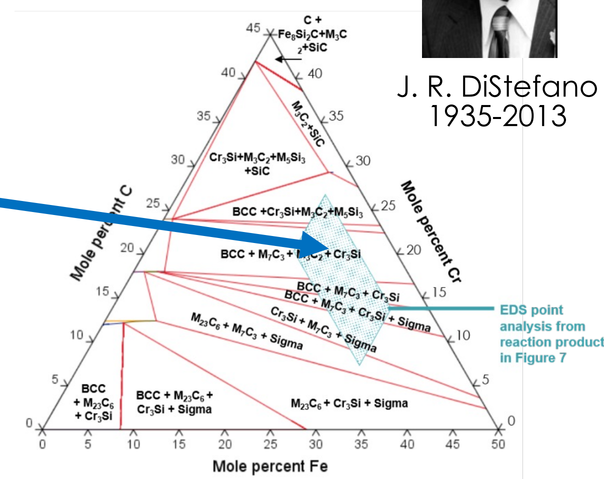


- Dissolved Fe & Cr in Pb-Li reacted with SiC to form carbides & silicides
- Accelerated dissolution of FeCrAl specimens
- Predicted by thermodynamics
  - After the experiment





J. R. DiStefano
1935-2013

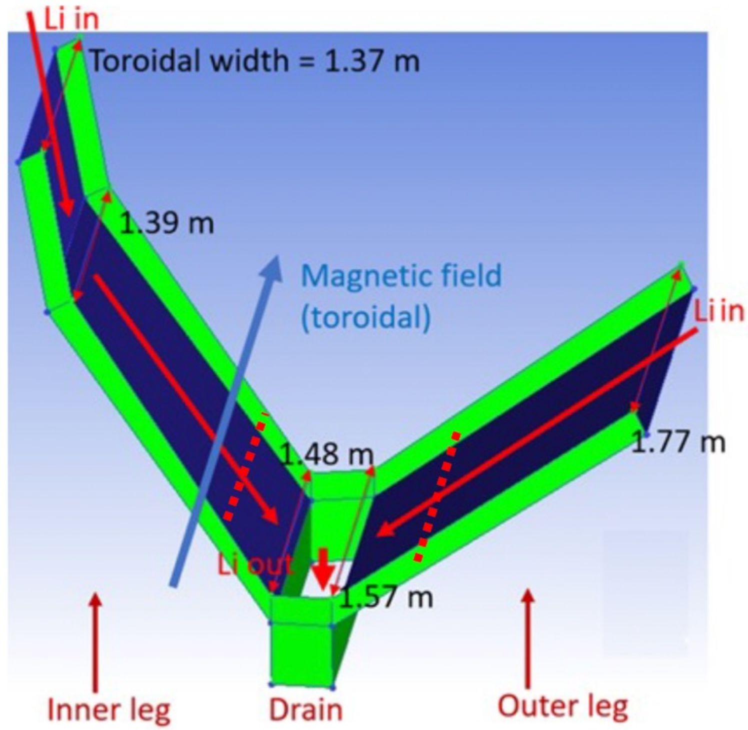


Pint, et al., Fusion Eng. Design, 166 (2021) 112389

Closing thoughts on ‘testing’ for fusion energy

- Material needs are going to be dependent on the concept
  - DCLL (Dual coolant PbLi) blanket is the current US focus
  - Many other blanket design options are being developed
  - General need for damage tolerant structural and functional materials
- Real ”testing” begins when we have a 14 MeV neutron source
  - Not my idea but I agree
- High temperatures are great (>1000°C)
  - But what is the working fluid and is it stable & compatible?
  - And how are you making tritium?
- Compatibility should not be ignored
  - but we sure haven’t made much progress in 70 years
- Outsiders: beware of ‘fusion forever’ perpetual research

Questions?



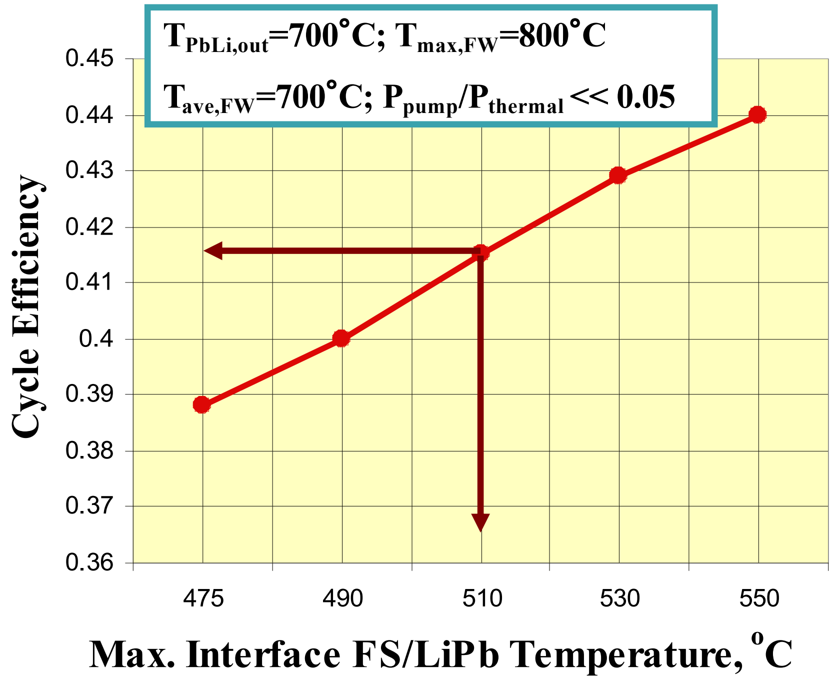
Kessel et al.: Liquid metal plasma facing component divertor design

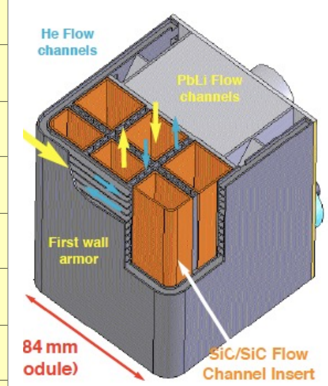
Can efficiency be improved with better steel-PbLi compatibility?

2005: Cycle Efficiency as a Function of Interface FS/Pb-17Li Temperature

UC San Diego

- For a fixed maximum neutron wall loading of ~4.7 MW/m^2:
  - max. η~38.8%, $T_{max,FS}$<<550°C for an interface temperature of 475°C
  - max. η~41.5%, $T_{max,FS}$<<563°C for an interface temperature of 510°C





FS = ferritic steel

475°C: typically accepted maximum Pb-Li compatibility temperature for 9Cr steel
550°C: Konys et al. (KIT, 2009 JNM) reported flowing Pb-Li loop plugged

LAMDA – Low Activation Material Development and Analysis Laboratory

Thermal/physical properties

Thermal expansion

Thermal transport

Electrical/ Seebeck

Mechanical properties

Torsion

Impact

Creep

High Temp Frames

Small Specimen Test Technology

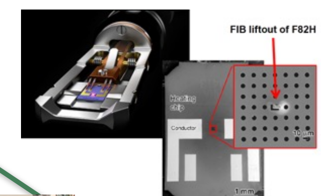
Microstructural characterization

Dual beam FIB

TEM

SEM/EBSD

STEM



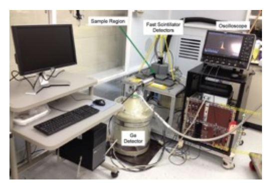
In-situ - various

LAMDA is a world-class, multipurpose **radiological materials science facility** for the evaluation of materials of low or no radioactivity. It consists of four laboratory suites containing specialized instrument for materials testing and characterization.

LECO O&H

GD-OES

Chemical analysis



Positron Annihilation

Thermal Desorption Spectrometry

Gas Permeability

Specialized instruments